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A LOOK AT TYPE K SHRINKAGE-COMPENSATING CEMENT PRODUCTION AND SPECIFICATIONS

by

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Concrete Laboratory
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Samples of Type K shrinkage-compensating cement from all 17 mills producing that cement in 1974 were obtained and evaluated for compliance with a proposed specification for these cements. The cements were also evaluated for specific gravity, fineness, heat of hydration, and expansion and drying shrinkage in mortars. Concretes were also made with the cements and evaluated for air content, slump, compressive strength, expansion, and drying shrinkage.

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In general most cements had little trouble meeting the specification requirements although a few cements had difficulty meeting the restrained-expansion requirements. The application of the proposed specification called attention to several shortcomings in the specification.

Both concretes and mortars made with Type K expansive cements generally had higher compressive strengths than comparably proportioned Type II cement mixtures. Slumps of Type K cement concrete were generally less than the Type II cement control mixture. Observations of air contents in both mortar and concrete suggest that some compatibility problems between certain individual cements and air-entraining agents may exist. The data indicate that the use of Type K cements in a wide variety of different applications should pose no extraordinary problems.

PREFACE

The work described in this Miscellaneous Paper was funded by the Office, Chief of Engineers, under work unit 010301/31140. The paper was prepared for presentation, cleared by the Office, Chief of Engineers, the Department of the Army, and the Department of Defense, and was presented at the Cedric Willson Symposium on Expansive Cements, during the American Concrete Institute meeting in New Orleans, Louisiana, in October 1977. The paper has been submitted to the American Concrete Institute for publication in the symposium volume.

The Concrete Technology Information Analysis Center (CTIAC) has provided funds to produce a small edition of this Miscellaneous Paper. This is CTIAC Report No. 29.

The paper was prepared by George C. Hoff and Katharine Mather, Chief, Materials Properties Branch, Engineering Mechanics Division (EMD), and Chief, Engineering Sciences Division, Concrete Laboratory (CL), Waterways Experiment Station (WES), under the general supervision of Messrs. J. M. Scanlon, Chief, EMD, and Bryant Mather, Chief, CL.

The Commander and Director of WES during the preparation and publication of this paper was COL J. L. Cannon, CE. Mr. F. R. Brown was Technical Director.

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A LOOK AT TYPE K SHRINKAGE-COMPENSATING CEMENT PRODUCTION AND SPECIFICATIONS[1]

by

George C. Hoff and Katharine Mather[2]

Synopsis: Samples of Type K shrinkage-compensating cement from all 17 mills producing that cement in 1974 were obtained and evaluated for compliance with a proposed specification for these cements. The cements were also evaluated for specific gravity, fineness, heat of hydration, and expansion and drying shrinkage in mortars. Concretes were also made with the cements and evaluated for air content, slump, compressive strength, expansion, and drying shrinkage. In general most cements had little trouble meeting the specification requirements although a few cements had difficulty meeting the restrained-expansion requirements. The application of the proposed specification called attention to several shortcomings in the specification.

Both concretes and mortars made with Type K expansive cements generally had higher compressive strengths than comparably proportioned Type II cement mixtures. Slumps of Type K cement concrete were generally less than the Type II cement control mixture. Observations of air contents in both mortar and concrete suggest that some compatibility problems between certain individual cements and air-entraining agents may exist. The data indicate that the use of Type K cements in a wide variety of different applications should pose no extraordinary problems.

Keywords: air content, compressive strength, drying shrinkage, expansive-cement concrete and mortar, expansive cement Type K, restrained expansion, shrinkage-compensating cements, slump tests, specifications, X-ray diffraction.

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1. Prepared for presentation at the Cedric Wilson Symposium on Expansive Cements, American Concrete Institute, New Orleans, LA, October 1977.

2. Chief, Materials Properties Branch, Engineering Mechanics Division, and Chief, Engineering Sciences Division, respectively, Concrete Laboratory, U. S. Army Engineer Waterways Experiment Station, Vicksburg, MS 39180.

INTRODUCTION

During the early 1970's there was considerable interest by several government agencies in using expansive cements in routine concrete construction as a means of minimizing shrinkage cracking. At that time there was no standard specification, however, for this type of cement by which an agency could insure the reliability and reproducibility of the product. Proposed specifications were being developed in Committee C-1 of the American Society for Testing and Materials (ASTM) but were not formally available for use. As a means of expediting the use of these cements in programs of the U. S. Army Corps of Engineers, an evaluation study was initiated in 1974 at the U. S. Army Engineer Waterways Experiment Station (WES), Vicksburg, Mississippi, to compare all of the Type K expansive cements in production at that time with the proposed ASTM specification for expansive cements which was in a draft form. The chemical and physical requirements of that proposed specification are contained in Table 1. Since that time the proposed specification has become ASTM C 845-76T, Tentative Specification for Expansive Hydraulic Cement,* but the requirements contained in Table 1 have not changed.

The purpose of the evaluation was two-fold. First was to test the adequacy and responsiveness of the proposed specification to the needs of the U. S. Army Corps of Engineers. Second, the evaluation would give an indication of the degree of variability that could be expected from the industry and would highlight any special problem areas that might limit the use of these cements.

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* 1977 Book of ASTM Standards, Part 13, pp 502-504.

MATERIALS

To make the necessary comparisons, a 100- to 200-lb (45- to 90-kg) sample of Type K shrinkage-compensating cement was obtained from the standard production runs of each of the 17 cement mills which were producing the cement at that time. A list of these mills is contained in Table 2. The samples were obtained over the period of March to December 1974 with most of the samples being obtained in April through August. In two instances, additional samples were obtained from a mill to substantiate the observed performance of the first sample. In these instances, there was no way to tell if the two samples came from the same production run. Each sample received was given a WES identification number.

TEST PROGRAM

Each cement was evaluated in accordance with the requirements of Table 1. In addition to these requirements, each cement was also evaluated for its specific gravity, fineness (air permeability), heat of hydration, and restrained drying shrinkage. An X-ray diffraction analysis was also made for each cement in an attempt to compare cements to note significant differences in composition or relative amounts of constituents. A standard concrete mixture was also developed in which each cement was used and evaluated for air content, slump, compressive strength, restrained expansion, and restrained drying shrinkage. The various test procedures used in these evaluations were as follows:

a. Chemical Analysis--ASTM Method C 114, Chemical Analysis of Hydraulic Cement.

b. Air Content--ASTM Method C 185, Air Content of Hydraulic Cement Mortar, using the actual specific gravity of the

cement if it differs from 3.15 by more than 0.05 in calculating the air content.

c. Time of Setting--ASTM Method C 807, Time of Setting of Hydraulic Cement Mortar by Vicat Needle.

d. Compressive Strength (Mortar)--ASTM Method C 109, Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens), except that a water-cement ratio of 0.5 was used, the specimens were covered with glass to prevent loss or gain of moisture at the surface of the specimens during the moist curing period in the molds, and the specimens remained in the molds for 3 days.

e. Restrained Expansion of Mortar--ASTM Method C 806, Restrained Expansion of Expansive Cement Mortar.

f. Specific Gravity--ASTM Method C 188, Specific Gravity of Hydraulic Cement.

g. Fineness--ASTM Method C 204, Fineness of Portland Cement by Air Permeability Apparatus.

h. Heat of Hydration--ASTM Method C 186, Heat of Hydration of Hydraulic Cement, determined at 7 and 28 days age.

i. Air Content (Concrete)--ASTM Method C 231, Air Content of Freshly Mixed Concrete by the Pressure Method, using a Type B meter.

j. Slump--ASTM Method C 143, Slump of Portland Cement Concrete.

k. Compressive Strength (Concrete)--ASTM Method C 39, Compressive Strength of Cylindrical Concrete Specimens, using 6-in. by 12-in. (152-mm by 305-mm) cylinders.

l. Restrained Expansion of Concrete--Proposed Test Method for Restrained Expansion of Shrinkage-Compensating Concrete,* except that all bars were demolded at 6-1/2 ± 1/4 hr with initial bar readings being made 30 minutes after demolding. All aggregate larger than 3/4-in. (19.0-mm) was wet screened from the mixture prior to casting the bars.

m. Drying Shrinkage--ASTM Method C 157, Length Change of Hardened Cement Mortar and Concrete, using both the restrained bars of mortar and concrete and, after 28 days moist curing, storage at 73.4 ± 3°F (23.0 ± 1.7°C) and 50 percent RH for an additional 90 days with length changes being noted to 90 days.

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* 1976 Annual Book of ASTM Standards, Part 14, pp 684-688.

MIXTURE PROPORTIONS

Mortar Mixture

The mortar mixture used for all the tests used one part of cement to 2.75 parts of graded Ottawa sand by weight. The water-cement ratio was 0.5 by weight. All mixing was done in a table-top paddle mixer. A Type II portland cement was also included in the test program for comparison purposes. Two rounds of test specimens were made for each cement.

Concrete Mixture

The concrete mixture used with all the expansive cements plus a Type II portland cement for control was as follows:

Ingredient	Saturated-Surface Dry Batch Weight, lb/yd^3 (kg/m^3)
Cement	550.0 (326.3)
Fine Aggregate	1146.8 (680.4)
Coarse Aggregate	
No. 4 - 3/4-in. (4.75 - 19.0 mm)	1071.3 (635.6)
3/4 - 1-1/2-in. (19.0 - 38.1 mm)	838.7 (497.6)
Water	291.5 (172.9)

The concrete was air entrained, and included 135 ml of air-entraining admixture per cubic yard. The coarse aggregate was crushed limestone. The fine aggregate was a manufactured limestone sand. The sand-aggregate ratio was 38 percent by volume. The water-cement ratio was 0.53.

TEST RESULTS FOR SPECIFICATION EVALUATION

The results of the chemical and physical tests of the cement in accordance with the expansive cement specification are contained in Tables 3 and 4, respectively.

Chemical Analyses

The results of the chemical analysis of 19 samples representing 17 mills (Table 3) show that all of the expansive cements meet the requirements for magnesium oxide (MgO), insoluble residue, and loss on ignition. Two mills (three samples, RC-694, -695, -695(2)) did not meet the optional requirement of 0.60 percent total alkalies with both mills exceeding the maximum allowable. The sulfate content (SO_3) of each cement, although not a specification requirement, is also shown in Table 3 as additional information.

The range of MgO values obtained was 0.7 to 4.3 percent with an average value of 2.2 percent. The insoluble residue content varied from 0.10 to 0.82 percent with an average value of 0.38 percent. The loss on ignition ranged from 0.9 to 2.4 percent with an average value of 1.8 percent. The optional requirement for total alkalies varied from 0.14 to 0.75 percent with an average value of 0.47 percent for all samples and ranged from 0.14 to 0.58 percent with an average value of 0.42 percent for those samples which met the specification requirements. The sulfate content ranged from 4.6 to 7.5 percent with an average value of 6.0 percent.

Air Content (Mortar)

The air content of the standard mortar mixture (Table 4) ranged from 7.3 to 10.9 percent with an average value of 8.7 percent. Each air content shown in Table 4 represents the average of measurements on two batches.

Compressive Strength (Mortar Cubes)

The compressive strength of the mortar cubes made with a given cement was determined from 2-in. (50-mm) cubes from two individual batches made with that cement. Each strength value shown in Table 4 represents the

average of six individual cube tests. For all 19 cement samples, the 7-day cube strengths ranged from 3070 psi (21.2 MPa) (RC-690) to 4730 psi (32.6 MPa) (RC-695) with an average strength of 4145 psi (28.6 MPa). The 28-day cube strengths ranged from 4860 psi (33.5 MPa) (RC-709) to 6500 psi (44.8 MPa) (RC-703) with an average strength of 5750 psi (39.6 MPa).

Restrained Expansion (Mortar Bars)

The restrained expansion values shown in Table 4 represent the average expansion of four bars, two each, from two batches made with each cement. The 7-day restrained expansion ranged from 0.028 (RC-687) to 0.104 percent (RC-690) with an average of 0.052 percent. Seven samples representing 6 mills or approximately 35 percent of the mills did not meet the 7-day expansion requirements. Initially 5 samples (RC-687, -694, -695, -700, -711) did not meet the minimum expansion requirement of 0.04 percent while 1 sample (RC-690) exceeded the maximum requirement of 0.10 percent. Upon evaluating a second sample from two of these mills, one of the mills with low value (RC-695) achieved satisfactory expansions while the mill with high value (RC-690) traversed the entire acceptable range of expansions and then failed the minimum expansion requirement (RC-690(2)).

The 28-day expansions were not to exceed the 7-day expansions by more than 15 percent. All but four (RC-690(2), -703, -709, -711) of the samples achieved this. The range of percentages of 7-day expansions was from 93 to 155 percent with an average change of 108 percent.

Time of Setting

Times of setting were determined for each cement on each of the two batches used to make the strength cubes and expansion bars. The times of setting ranged from 1 hour 40 minutes to 4 hours 50 minutes with an

average time of setting of 2 hours 50 minutes.

Other Cement Data

Although not specifically called for in the expansive-cement specification, additional data on characteristics of the expansive cements were obtained to aid in the overall evaluation of these cements. These characteristics included specific gravity, fineness, and heat of hydration (Table 3).

The specific gravity ranged from 3.00 to 3.17 with an average value of 3.09. The fineness (air permeability) ranged from 3550 to 5360 cm^2/g with an average fineness of 4350 cm^2/g. The heat of hydration at 7-days age ranged from 63 to 88 cal/g with an average value of 75 cal/g, while at 28-days age the range increased to 77 to 96 cal/g with an average value of 88 cal/g.

CONCRETE TEST RESULTS

The expansive-cement concrete portion of this study was done to get an indication of any variability effects the cements from across the entire industry might have on such concrete characteristics as air content, slump, compressive strength, and restrained expansion. The proportions of each batch were the same with only the cement being varied. The results of the physical tests of the concrete are contained in Table 5. Each result represents the average of test specimens from two batches of concrete. The restrained-expansion bars were made from concrete which was wet-sieved over a 3/4-in. (19.0-mm) sieve. This was necessitated by the maximum size of the aggregate (1-1/2-in. (38.1-mm) MSA), the least dimension of the bar (3-in. or 76-mm), and the centrally located restraining rod in the bar.

Air Content (Concrete)

The air content of the concrete varied for the individual batches from 2.2 percent for RC-698 to 6.0 percent for RC-700. The average air content for concrete made with all 19 cements was 4.1 percent. The Type II control mixture had an average air content of 3.8 percent.

Slump

The slump of the individual batches of concrete varied from a low of 2-1/4 in. (57 mm) for RC-702. The average slump for all the expansive cement concrete rounded to the nearest 1/8 in. (3.2 mm) was 3-1/4 in. (83 mm). The average slump of the Type II control concrete was 3-7/8 in. (98 mm).

Compressive Strength (Concrete Cylinders)

The compressive strength of the concrete was determined from testing 6-in. by 12-in. (152-mm by 305-mm) cylinders. Each batch of concrete had two cylinders evaluated at both 7 and 28 days age. The values shown in Table 5 represent the average of two batches for each cement or hence four cylinders evaluated at each age.

The 7-day compressive strengths of the individual batches varied from 2970 psi (20.5 MPa) for RC-709 to 4740 psi (32.7 MPa) for RC-707. The average 7-day strength for all 19 cements was 3830 psi (26.4 MPa). The Type II control strength at the same age was 3410 psi (23.5 MPa). The 28-day strengths varied from 4250 psi (29.3 MPa) for RC-709 to 6130 psi (42.2 MPa) for RC-695(2). The average 28-day strength for all 19 cements was 5130 psi (35.4 MPa). The Type II control strength at the same age was 4700 psi (32.4 MPa).

Restrained Expansion (Concrete Bars)

The restrained-expansion values shown in Table 5 represent the average expansion of four bars, two each from two batches made with each cement. The 7-day restrained expansions ranged from 0.024 percent for RC-687 to 0.130 percent for RC-690 with an average restrained expansion of 0.044 percent. The 28-day restrained expansion ranged from 0.027 percent for RC-687 to 0.133 percent for RC-690. It should be noted that both these cements also established the range limits for the 7-day expansions. The average 28-day restrained expansion for all 19 cements was 0.045 percent which was not significantly different than the average expansion at 7 days.

The average restrained expansions for the Type II control mixture at 7 and 28 days were -0.002 percent and +0.002 percent, respectively.

DRYING SHRINKAGE TEST RESULTS

Drying shrinkage of cement paste, mortar, and concrete is usually measured on unrestrained prisms of the material. Restraint is the mechanism by which expansive cements effectively use their expansions, however, this restraint should also then be present when the expansive cements begin to lose their effectiveness through drying. The data contained in Tables 6 and 7 represent the restrained drying shrinkage of the same restrained expansion prisms used for the mortars and concretes, respectively. Although the prisms were allowed to dry for 90 days, most of the length change had occurred after 60 days of drying. The shrinkage values reported in both Tables 6 and 7 are referenced to the length of each bar after 28-days expansion.

Restrained Mortar Bar Shrinkage

The length changes of the restrained mortar bars after 90 days of curing at 73°F and 50 percent relative humidity shown in Tables 6 and 7 varied from a low value of 0.065 percent for RC-695(2) to a high value of 0.103 percent for RC-709. The average shrinkage for all 19 cements was 0.080 percent, which was slightly more than the 0.063 percent shrinkage experienced by the Type II control cement.

When these shrinkage values are combined with the observed expansions which preceded the shrinkage, the net change in bar lengths was observed to vary from a net increase of 0.007 percent for RC-707 to a net decrease of 0.044 percent for RC-695. The average net length change for all cements was a shrinkage of 0.028 percent as compared to a net shrinkage of 0.058 percent for the Type II portland-cement control.

Restrained Concrete Bar Shrinkage

The length changes of the restrained concrete bars after 90 days of curing at 73.4°F (23.0°C) and 50 percent relative humidity are shown in Table 7 and varied from a low value of 0.033 percent for RC-707 to a high value of 0.052 percent for RC-691. The difference in the high and low values for the concrete was 0.019 percent, which was one-half the difference observed for the mortar. The average shrinkage for all 19 cement samples was 0.044 percent, which was not significantly different than the 0.045 percent shrinkage observed for the Type II portland-cement control bars.

Combining these shrinkage values with the observed expansions which preceded the shrinkage, the net change in bar lengths was observed to vary from a net increase of 0.082 percent to a net decrease of -0.025 percent for RC-687. The range of these length changes was approximately

twice the range observed for the mortar bars. Average net length change for all the cements was 0 percent as compared to a net shrinkage of 0.043 percent for the Type II portland-cement control.

THE CONSTITUTION OF THE CEMENTS

X-Ray Diffraction Test Procedures

Each cement was examined in the as-received condition, front loaded and tight packed in a sample holder giving a sample surface about 2-3/4 in. (70 mm) long. All examinations were made in a static nitrogen atmosphere. Another sample of each cement, weighing 5.0000 g, was treated with maleic acid in absolute methanol to remove the calcium silicates and leave calcium aluminoferrites, tricalcium aluminate (C_3A), calcium sulfates, tetracalcium trialuminate sulfate ($C_4A_3\bar{S}$), and magnesia (MgO) in the insoluble residue. The residue was weighed after drying to a free-flowing condition and was examined on the diffractometer. A few of the residues insoluble in maleic acid were treated with 10 percent ammonium chloride (NH_4Cl) solution to remove calcium sulfates and were then examined on the diffractometer.

The diffractometer was standardized using an external quartz standard before each examination. Although the X-ray diffraction work covered a period of 10 months the diffraction charts are regarded as comparable because of the standardization and adjustment of the ratemeter, kilovoltage (kv), and milliamperage (ma) to comparable intensity levels at low-power and high-power settings. Low power was used from 5 to 20° two theta, with 27 kilovolts constant potential (kvcp) and 41 ma, using a 1° beam slit, 3° beam slit as a soller slit, and a 0.2° detector slit. The chart range was approximately logarithmic from 10 to 4000 counts for full-scale deflection.

High-power settings were used for the range from 20° two theta to 65°, at 50 kvcp and 21 ma, using a 3° beam slit and the same scale, with the rest of the X-ray collimation as for low power. All of the X-ray diffractometer charts were compared and intensities in chart units were measured on the charts of as-received cements and of maleic acid-insoluble residues, after a background had been drawn to bisect the background level from 20° to 65° two theta and sketched from 5° to 20° two theta on the diffraction charts. Table 8 shows the most easily measured intensities of alite (substituted tricalcium silicate), belite (substituted dicalcium silicate), the strongest line of $C_4A_3\overline{S}$, two tricalcium aluminate lines, the strongest line of magnesia (MgO), and an aluminoferrite line of fairly high intensity that is not interfered with.

It is possible that some of the maleic acid used to remove silicates in fact contained other materials that also affected other constituents, because some of the nominal maleic acid was not satisfactory in determining cement in hardened concrete.

Identification of Constituents

Calcium sulfate was found in these cements as $CaSO_4$ (anhydrite), $CaSO_4 \cdot 0.5H_2O$ (plaster of paris), and $CaSO_4 \cdot 2H_2O$ (gypsum). In 10 of the cements gypsum was shown clearly to be present and made a considerable contribution to the total calcium sulfate. In four it was barely detectable; and in the remaining five it was not detected by X-ray diffraction in the as-received samples. All 19 cements contained $CaSO_4$ (anhydrite). All of the cements contained $CaSO_4 \cdot 0.5H_2O$ after treatment with maleic acid, but it was not surely identified, although it was suspected in the diffraction charts in several of the cements as received. Because of

the uncertainty as to identifying $CaSO_4 \cdot 0.5H_2O$ in the as-received cements, because of the distribution of calcium sulfate in two or three compounds in most of the cements, and since each sulfate differs from the others in absorption coefficient and thus somewhat in diffracting ability, there was no satisfactory measurement of total calcium sulfate by X-ray diffraction and Table 8 shows SO_3 by chemical analysis as a measure of calcium sulfate. Tricalcium aluminate (C_3A) was identified in 17 of the 19 cements.

Effects of Composition

Although C_3A as well as $C_4A_3\bar{S}$ can participate in forming ettringite, and although C_3A was as abundant as or more abundant than $C_4A_3\bar{S}$ in several of the cements, it appears that the C_3A did not affect the restrained expansion significantly with the possible exception of RC-707, which contained more C_3A than any of the other cements. The restrained expansion at 7 days was significantly higher than in several other cements with similar intensity of $C_4A_3\bar{S}$ but RC-707 also contained more SO_3 than any of the others. It seems reasonable to conceive that $C_4A_3\bar{S}$ blended with portland cement is more easily accessible to hydration and formation of ettringite than C_3A which may be protected in part from hydration by other cement constituents and thus hydrate more slowly to, or convert to, monosulfate ($C_4A\bar{S}H_{12}$) rather than forming and persisting as ettringite. The apparent absence of effect of C_3A in these shrinkage-compensating cements was somewhat surprising.

Aluminoferrite is recorded in Table 8 but no effect on the behavior of the shrinkage-compensating cements was expected from it or found.

The influences on restrained expansion that appear to be most active

include the amount of $C_4A_3\bar{S}$ (as indicated by the height of the line at 3.76 A), the fineness of the cement, the amount of alite, and the total amount of sulfate. Strength at early ages is also significant, and extremes of fineness. Examples are given in the discussion that follows. RC-687 had the lowest fineness recorded, low $C_4A_3\bar{S}$, high alite; the 7-day compressive strength was above average, which all suggests that there was too little $C_4A_3\bar{S}$ to form enough ettringite to expand the fairly strong framework developed in the first days of strength gain. Six cements in the group failed to meet ASTM specification requirements for expansion of 0.040 percent at 7 days (RC-687, -690(2), -694, -695, -700, -711); all had $C_4A_3\bar{S}$ intensity below 24, alite ranging from 27 to 43, SO_3 from 5.2 to 6.4 percent, fineness ranging from 3550 to 5360 cm^2/g (the highest recorded), and 7-day strengths from 3840 to 4730 psi, the latter being the highest 7-day strengths in the group. On the other hand, two cements with $C_4A_3\bar{S}$ intensities of 19 and 21 met the 7-day expansion requirement; one met it barely and had low total sulfate (5.1 percent) and low alite. The other had the highest sulfate of any of the cements and moderately high alite. RC-690, which had the highest expansion at 7 days had rather low alite and the lowest 7-day strength in the group. Four cements had expanded at 28 days more than the allowed 115 percent of the 7-day expansion; one had the highest $C_4A_3\bar{S}$ and alite close to the average; three had average $C_4A_3\bar{S}$ and alite average or below.

$C_4A_3\bar{S}$ intensities from 23.5 to 29 with alite intensities ranging from 30 to 41 associated with finenesses from 3730 to 5240 cm^2/g produced cements that met the requirements of the ASTM specification; the sulfate contents in the group of acceptable cements ranged from 4.6 to 7.5 percent.

The regression equation using amount of $C_4A_3\bar{S}$ as X and the 28-day restrained expansion as Y is a straight line function of the form

$$Y = -0.0214 + 0.0032\ X \quad (1)$$

The correlation coefficient is $R = +0.56$. For $n - 2 = 17$, this correlation would occur by chance less than 2 times in 100.[1] Considering that the behavior in restrained expansion of these cements must be affected by compressive and tensile strength of the specimens, which depends on fineness, cement composition, and sulfate available to provide early strength, it is somewhat surprising to find a significant relation between these two variables. The correlation of SO_3 and restrained expansion at 7 days was calculated but was just below significant at the 5 percent level.

A regression equation was calculated between MgO determined chemically (X) and MgO in X-ray diffraction chart units (Y) and was best represented by a simple power function of the form

$$Y = 4.4X^{1.37} \quad (2)$$

with a correlation coefficient of +0.94, which for $n - 2 = 17$ has substantially less than one chance in 100 of the relation occurring by chance (Fig. 1).

CaO (free lime), a significant constituent of self-stressing cements, was not certainly identified in any of the cements of this group. While it may have been present in some of the cements in small amounts, it appears that the Ca needed to combine with $C_4A_3\bar{S}$ and $CaSO_4$ to form ettringite was obtained as $Ca(OH)_2$ from the hydration of alite.

The means, standard deviations, and variances of several variables are shown in Table 9.

From the examinations by X-ray diffraction it seems probable that five of the shrinkage-compensating cements have their portland-cement portion low in C_3A; sufficiently so as to have this portion regarded as like Type V portland cement. In these five, no C_3A line was detected at 1.558 A. Possibly the portland-cement portion of a few others had Type II compositions. Several apparently contained Type I portland cements. The factors on which the behavior of these Type K shrinkage-compensating cements depend appear to be more difficult to assess than was the case with 15 self-stressing cements examined previously.[2]

DISCUSSION OF RESULTS

Specification Compliance

None of the cements had any difficulty meeting the mandatory chemical requirements of the specifications although cements from two mills did not meet the optional maximum requirement for total alkalies.

None of the cements appeared to have a problem meeting the physical requirements of air content, compressive strength, and time of setting. There was a problem with the principal feature for which these cements were designed however, that is expansion, with several cements not meeting the requirements for 7-day expansion. Of the initial sampling, five cements did not meet the minimum expansion requirements, having values 0.028, 0.034, 0.033, 0.039, and 0.037 percent. A review of these values together with the 0.036 percent value obtained from the second sample of one mill (RC-690(2)) points out a feature of the specification which warrants further definition.

The length change of the restrained bars is measured in a comparator

to the nearest 0.0001 in. (0.0025 mm). Expressed as a percentage of length change, measurements are then made and reported to the nearest 0.001 percent, which is a number of three significant figures. The minimum and maximum expansion limits of the specification are 0.04 percent and 0.10 percent, respectively, which are numbers of two significant figures. Having made measurements to three significant figures, they are then rounded to two significant figures for compliance to the specification. The result of the rounding is that three of the six cements which failed the minimum expansion requirement (RC-690(2), -700, and -711) and the one cement which failed the maximum expansion requirement (RC-690) at three significant figures, now meet the specification at two significant figures. On this point, the intent of the proposed specification is not clear. To eliminate this dilemma and insure that cements with adequate and safe expansive potentials are being used, the limits of expansion in the specification probably should be expressed to three significant figures as 0.040 percent and 0.100 percent for minimum and maximum limits, respectively.

The problem of two versus three significant figures also carries over into the requirement that the 28-day expansions be not more than 115 percent of the 7-day expansions. Using the three significant figures obtained in the actual length change measurements, four cements (RC-690)2), -703, -709, and -711) failed this requirement. If the numbers had been rounded to two significant figures prior to determining the increase, only three of those four cements would have failed. The proposed specification should contain more explicit language as to how this increase in percentage is to be determined.

The allowable later expansions expressed as a 115 percent increase should also be reviewed from the standpoint of the actual expansions that occur. Under this requirement, cements expanding to the minimum level of 0.04 percent or 0.004 in. (0.102 mm) for a 10-in. (254-mm) restrained bar, would be allowed to increase in length by an additional 0.0006 in. (0.0152 mm). At the maximum allowable level of 0.10 percent, or 0.010 in. (0.254 mm) for a 10-in. (254-mm) restrained bar, the acceptable increase could be 0.0015 in. (0.0381 mm) or 2-1/2 times the increase of the bar at the minimum level. For two concretes of identical proportions, all other things also being equal, except for the expansive potential of the cement, a concrete having a large initial expansion (0.010-in. (0.0254-mm)) will probably not tolerate an additional expansion of 0.0015-in. (0.0381-mm) as well as would a concrete having a low initial expansion (0.004-in. (0.102-mm)). This is not to say that an additional expansion of 0.0015-in. (0.0381-mm) would be harmful to a concrete expanding 0.10 percent. This fact is not known to the authors but if in fact it is not harmful, that amount of additional increase could also be safely tolerated by a concrete expanding 0.04 percent at 7 days age. This suggests that the additional expansion requirement should perhaps be stated as a fixed amount over the entire range rather than as a percent increase. The amount of this fixed value would have to be defined and substantiated by additional testing.

The X-ray diffraction analysis indicates that the performance of the cements with regard to expansion is affected by the amounts and types of constituents not identified in the chemical requirements. This suggests that chemical requirements might be expanded to include other chemical requirements that may provide a more rapid indication of the expansive

potential of the cements. These additional requirements might include upper and lower limits on SO_3 and Al_2O_3.

Expansive Potential Requirements

The principal reason for using expansive cements in concrete is, of course, to provide an overall combination of expansion and drying shrinkage such that the resulting dimensions of the element made with the concrete will not cause tensile stresses of such magnitude so as to cause cracking to occur within the element. The data in Table 6 indicate that after 90 days restrained drying shrinkage, all but two of the cements had shrunk to less than the original length of the test specimens before expansion had occurred. The average change from initial length was a shrinkage of 0.028 percent. With additional time in the same curing environment, even more shrinkage will take place. Even the concrete (Table 7), which had less cement per unit volume to contribute to shrinkage and which also had its shrinkage restrained by the presence of coarse aggregate, had 10 of the 19 cements exhibit more shrinkage than expansion. Although not measured, the shrinkage trends indicated that by 120 days age, 16 of the 19 cements would have had more shrinkage than expansion.

It is not known whether these values of shrinkage for either the mortar or concrete would induce shrinkage cracking in a concrete element because of the many factors which can influence shrinkage in concrete. They do suggest however that if volume stability (zero length change after expansion and shrinkage) will insure the elimination or significant reduction in drying shrinkage cracking. The minimum specification limit of 0.04 percent may not be adequate and perhaps should be increased. The amount of this increase would have to be determined through additional testing.

Other Cement Characteristics

The specific gravities of the expansive cements were generally less than those normally associated with Type I and II portland cements although a few exceptions occurred. This presents no problem to their use except that this fact should be called to the attention of the cement user so he will make the necessary determinations of this value before beginning mixture proportioning.

The fineness of the expansive cements is generally higher than that of Type I and II portland cements and is comparable to those normally associated with Type III portland cements. These higher fineness values may be the result of the increased amount of gypsum in these cements. The gypsum, being softer than cement clinker, will reduce more readily during grinding and thus increase the apparent fineness of the cement.

The heats of hydration of Type K expansive cements apparently are not significantly different from the usual portland cements and thus could be used in larger concrete sections without increasing thermal problems.

The fact that air contents in concrete varied from 2.4 to 6.0 percent for the same amount of a given brand of air-entraining admixture in each concrete batch and varied from 7.2 to 11.3 percent in mortar suggests that there is a compatibility requirement for air-entraining admixtures and expansive cements which may warrant further study.

The slumps of concretes made with Type K expansive cements have historically been reported as always being less than comparably proportioned Type I and II portland-cement concrete mixtures. This has been attributed to both increased cement fineness and the need for additional water in the formation of ettringite. In general, most of the cements did not produce

a lower slump than the Type II cement control; however, some exceptions were noted.

As with slumps, compressive strengths of Type K expansive cements have historically been reported as being more than those achieved with comparable proportioned mixtures made with Type I and II cements. This has usually been attributed to the increased fineness of the Type K cements, the reduction of free water in the concrete as more of the batch water is used for the development of hydration products, and also to the densification of the paste caused by the formation of larger amounts of ettringite. In general, the 19 cements evaluated exhibited this behavior, but again, there were some exceptions.

CONCLUSIONS

In assessing the significance of the data reported herein it must be appreciated that cements evaluated were from production in 1974 and do not necessarily reflect the condition of the Type K expansive cement industry at the time this paper was published. What the data do indicate, however, are some apparent shortcomings in the proposed specification for these cements. The proposed specification, as presently written, may satisfy most user requirements; however, there are a number of aspects of it which could be revised to form a much stronger and responsive specification. These include (1) use of additional chemical requirements to assess expansive potential more quickly, (2) a restatement of expansion requirements to three significant figures, (3) an upgrading of the minimum expansion requirement of the specifications, and (4) the revision of the later age expansion requirement to a constant value requirement rather

than a percentage requirement.

When moist cured, Type K expansive cements do in fact expand substantially more than normal portland cements. When they undergo drying shrinkage, the amount of that shrinkage is similar to that of normal portland cements. The combined expansion and shrinkage produce less total shrinkage in test prisms than occurs with normal portland cements.

The other characteristics of the Type K expansive cements such as specific gravity, fineness, and heats of hydration present no special problems for their use in a wide variety of different applications. When used in concrete they tend to reduce slumps and slightly increase compressive strengths at 28-day ages over those of comparably proportioned Type I and II cement concretes. There may be some slight compatibility problems between individual cements and air-entraining agents, but these should be evaluated on an individual basis.

The tests described and the resulting data presented herein, unless otherwise noted, were obtained from research conducted under the Civil Works Research Program of the United States Army Corps of Engineers by the U. S. Army Engineer Waterways Experiment Station. Permission was granted by the Chief of Engineers to publish this information.

REFERENCES

1. Fisher, R. A., Statistical Methods for Research Workers, 10th ed., G. E. Stechert and Co., New York, N. Y., 1946, pp 183-209.

2. Mather, Katharine, "X-Ray Diffraction Examination of the Phases in Expansive Cements," Advances in X-Ray Analysis, Vol. 20, Plenum Publishing Corp., New York, N. Y., 1977.

Table 1

Tentative ASTM Specification C 845-76T

Chemical	
Magnesium Oxide (MgO), %	5.0 (Max)
Insoluble Residue, %	1.0 (Max)
Loss on Ignition, %	4.0 (Max)
Total Alkalies, % (Na_2O + 0.658 K_2O)	0.60 (Max)
Physical	
Air Content, %	12.0 (Max)
Compressive Strength, psi (MPa)	
7-Day	2100 (14.7) (Min)
28-Day	3500 (24.5) (Min)
Restrained Expansion, %	
7-Day	0.04 (Min) To 0.10 (Max)
28-Day	115 (Max) Of 7-Day Expansion
Time of setting, minutes	90 (Min)

Table 2

Type K Cements Evaluated

Texas Industries, Inc.
Midlothian, Texas
Southwestern Portland Cement Co.
Odessa, Texas
Fairborn, Ohio
Penn-Dixie Cement Corp
Petoskey, Michigan
Kingsport, Tennessee
West Winfield, Pennsylvania
Nazareth, Pennsylvania
Howes Cave, New York
West Des Moines, Iowa
Medusa Portland Cement Co.
York, Pennsylvania
Clinchfield, Georgia
Wampum, Pennsylvania
Dixon, Illinois
Kaiser Cement and Gypsum Corp
Permanente, California
Lucerne Valley, California
San Antonio, Texas
Montana City, Montana

Table 3

Summary of Expansive Cement Chemical Test Data

Constituent	Tentative ASTM Specification	Type II Control	RC-687	RC-689	RC-690	RC-690(2)	RC-691	RC-692	RC-693	RC-694	RC-695
Magnesium Oxide (MgO), %	5.0 (Max)	1.0	0.7	0.8	3.9	4.3	3.1	2.9	1.0	2.1	2.2
Insoluble Residue, %	1.0 (Max)	0.12	0.59	0.10	0.45	0.28	0.22	0.15	0.82	0.33	0.52
Loss on Ignition, %	4.0 (Max)	0.8	1.3	0.9	2.2	2.0	2.1	1.4	2.1	2.3	2.2
Total Alkalies, % (Na_2O + 0.658 K_2O)	0.60(Max)	0.54	0.43	0.27	0.58	0.56	0.45	0.57	0.38	0.77	0.75
SO_3, %	--	2.0	5.9	4.6	6.8	5.0	6.0	6.0	6.3	6.4	5.9

Table 3 (Cont)

Constituent	Tentative ASTM Specification	Type II Control	Type K Expansive Cement									
			RC-695(2)	RC-696	RC-697	RC-698	RC-700	RC-702	RC-703	RC-707	RC-709	RC-711
Magnesium Oxide (MgO), %	5.0 (Max)	1.0	2.2	2.8	3.6	2.4	1.1	0.9	1.9	1.1	2.7	2.9
Insoluble Residue, %	1.0 (Max)	0.12	0.39	0.37	0.23	0.60	0.18	0.35	0.54	0.17	0.42	0.48
Loss on Ignition, %	4.0 (Max)	0.8	2.3	2.4	1.6	2.1	1.7	1.1	1.1	1.8	1.6	2.3
Total Alkalies, % (Na_2O + 0.658 K_2O)	0.60(Max)	0.54	0.70	0.43	0.45	0.14	0.46	0.48	0.40	0.26	0.37	0.56
SO_3, %	--	2.0	6.1	5.8	5.1	6.3	6.0	6.3	6.5	7.5	6.7	5.2

Table 4

Summary of Expansive Cement Physical Test Data

	Tentative		Type K Expansive Cement							
Physical Property*	ASTM Specification	Type II Control	RC-687	RC-689	RC-690	RC-690(2)	RC-691	RC-692	RC-693	RC-694
Air Content, %	12.0 (Max)	8.1	9.0	8.8	8.8	8.3	9.3	7.2	7.7	10.0
Time of Setting, Hr:Min	1:30(Min)	--	3:00	1:40	2:00	2:00	3:00	2:55	2:40	2:50
Compressive Strength, psi										
7-Day	2100 (Min)	3160	4320	4500	3070	4190	4240	4680	4320	4560
28-Day	3500 (Min)	--	5700	5550	4940	5470	5690	6020	5970	5720
Restrained Expansion,%										
7-Day	0.04 To 0.10	0.004	0.028	0.058	0.104	0.036	0.058	0.046	0.058	0.034
28-Day	Not more than 115 % of 7-Day Expansion	125	100	109	102	125	102	100	96	103
Specific Gravity	---	3.14	3.09	3.13	3.06	3.09	3.06	3.11	3.08	3.05
Fineness, Blaine (Air Permeability, cm^2/gm)	---	3295	3550	3730	4290	4220	4210	4220	4330	4510
Heat of Hydration, cal/gm										
7-Day	---	--	80	85	72	78	77	75	72	88
28-Day	---	--	87	96	89	88	97	86	92	94

* All values are the average of specimens from two rounds of mortar.

Table 4 (Cont)

Physical Property*	Tentative ASTM Specification	Type II Control	Type K Expansive Cement										
			RC-695	RC-695(2)	RC-696	RC-697	RC-698	RC-700	RC-702	RC-703	RC-707	RC-709	RC-711
Air Content, %	12.0 (Max)	8.1	8.6	7.6	10.9	11.3	10.1	7.3	8.4	7.7	7.8	9.3	7.8
Time of Setting, Hr:Min	1:30(Min)	--	2:12	2:10	3:10	3:06	2:29	2:40	4:50	2:35	4:27	4:48	2:02
Compressive Strength, psi													
7-Day	2100 (Min)	3160	4730	4470	3880	4170	4530	4230	3570	3960	4320	3220	3840
28-Day	3500 (Min)	--	6010	--	5080	5190	6600	5740	5990	6500	7010	4860	5290
Restrained Expansion,%													
7-Day	0.04 To 0.10	0.004	0.033	0.057	0.065	0.040	0.046	0.039	0.055	0.063	0.068	0.062	0.037
28-Day	Not more than 115 % of 7-Day Expansion	125	100	93	98	102	100	108	104	119	107	155	132
Specific Gravity	---	3.14	3.05	3.08	3.00	3.12	3.09	3.11	3.16	3.17	3.06	3.12	3.09
Fineness, Blaine (Air Permeability, cm^2/gm)	---	3295	5360	4890	4500	3600	5240	4210	4520	4490	4180	4270	4370
Heat of Hydration, cal/gm													
7-Day	---	--	84	--	79	75	67	71	67	70	73	63	73
28-Day	---	--	88	--	84	84	83	84	81	83	94	77	85

* All values are the average of specimens from two rounds of mortar.

Table 5

Summary of Expansive Cement Concrete Test Results

Physical Property*	Type II Control RC-658	Type K Expansive Cement RC-687	RC-689	RC-690	RC-690(2)	RC-691	RC-692	RC-693	RC-694	RC-695
Slump, in.	3 7/8	4 1/8	4 1/8	2 7/8	2 1/2	3	3	3	3 1/4	3
Air Content, %	3.8	4.8	4.6	3.4	4.0	3.6	2.4	3.3	3.9	2.6
Unit Weight, pcf	147.1	144.3	146.7	147.0	147.9	147.1	148.7	147.5	145.8	148.1
Compressive Strength, psi										
7-Day	3335	3605	3595	3535	3970	4000	4185	3860	4205	4145
28-Day	4645	4665	4910	4665	5015	5205	5565	5080	5180	5465
Restrained Expansion, %										
7-Day	-0.002	0.024	0.065	0.130	0.039	0.047	0.034	0.028	0.030	0.050
28-Day	0.002	0.027	0.070	0.133	0.040	0.046	0.033	0.030	0.030	0.051

* All values are the average of specimens from two rounds of concrete.

Table 5 (Cont)

Physical Property*	Type II Control	Type K Expansive Cement									
	RC-658	RC-695(2)	RC-696	RC-697	RC-698	RC-700	RC-702	RC-703	RC-707	RC-709	RC-711
Slump, in.	3 7/8	2 3/4	2 1/2	3 1/2	2 3/8	3 3/4	4 1/2	3 1/2	4	4 1/8	3 3/8
Air Content, %	3.8	3.4	2.9	5.3	2.4	6.0	5.2	5.5	4.2	5.2	4.8
Unit Weight, pcf	147.1	149.6	148.2	144.6	148.4	145.6	146.2	146.2	148.4	147.1	144.2
Compressive Strength, psi											
7-Day	3335	4085	4455	3560	4080	3630	3355	3405	4560	3135	3465
28-Day	4645	5865	5820	4470	5845	4580	5135	5160	5720	4495	4710
Restrained Expansion, %											
7-Day	-0.002	0.044	0.037	0.028	0.032	0.025	0.044	0.052	0.036	0.038	0.029
28-Day	0.002	0.050	0.041	0.030	0.032	0.028	0.044	0.052	0.036	0.048	0.030

* All values are the average of specimens from two rounds of concrete.

Table 6

Summary of Length Change Measurements on Restrained Mortar Bars

WES Cement Designation	Mortar Bars: Total 28-Day Expansion, %	Total 90-Day Shrinkage, %	Change From Initial Length, %
Control	0.005	0.063	-0.058
687	0.028	0.079	-0.051
689	0.063	0.090	-0.027
690	0.106	0.092	+0.014
690(2)	0.045	0.071	-0.026
691	0.059	0.094	-0.035
692	0.046	0.083	-0.037
693	0.056	0.085	-0.029
694	0.035	0.083	-0.048
695	0.033	0.077	-0.044
695(2)	0.053	0.065	-0.012
696	0.064	0.097	-0.033
697	0.041	0.071	-0.030
698	0.046	0.086	-0.040
700	0.042	0.073	-0.031
702	0.057	0.073	-0.016
703	0.075	0.084	-0.009
707	0.075	0.066	+0.007
709	0.096	0.103	-0.007
711	0.049	0.072	-0.023
Average	0.056	0.080	-0.028

Table 7

Summary of Length Change Measurements on Restrained Concrete Bars

WES Cement Designation	Concrete Bars: Total 28-Day Expansion, %	Total 90-Day Shrinkage, %	Change From Initial Length, %
Control	0.002	0.045	-0.043
687	0.027	0.052	-0.025
689	0.070	0.050	+0.020
690	0.133	0.051	+0.082
690(2)	0.040	0.037	+0.003
691	0.046	0.052	-0.006
692	0.033	0.045	-0.012
693	0.030	0.047	-0.017
694	0.030	0.049	-0.019
695	0.051	0.047	+0.004
695(2)	0.049	0.044	+0.005
696	0.041	0.048	-0.007
697	0.030	0.040	-0.010
698	0.032	0.043	-0.011
700	0.028	0.041	-0.013
702	0.044	0.041	+0.003
703	0.052	0.035	+0.017
707	0.036	0.033	+0.003
709	0.048	0.043	+0.005
711	0.030	0.047	-0.017
Average	0.045	0.044	0.000

Table 8

X-Ray Diffraction Intensities in Chart Units, SO_3, and Amount Insoluble in Maleic Acid in Percent

WES Cement Designation	As Received		SO_3, %	Insoluble in Maleic Acid, % (By Weight)	Constituents Insoluble in Maleic Acid				
	Alite 1.76A[a]	Belite 2.88A[b]			$C_4A_3\bar{S}$[c] 3.76A	C_3A[d] 1.905A	C_3A[d] 1.558A	MgO[c] 2.10A	Aluminoferrites[c] 7.2-7.3A
RC-687	43	11	5.9	28.07	21.7	27	23	2.5	36
689	41	10	4.6	22.48	25.2	12	12	2	12
690	29	14	6.8	27.03	27.5	20	15	31	16
690(2)	34	8	5.0	27.45	22.5	15	15	35	12
691	39	11	6.0	26.06	26	26	19	18	12
692	31	13	6.0	28.01	24.5	22	16	12	14
693	35	11	6.3	26.50	23.5	18	11	6	18
694	29	7	6.4	31.78	23	29	23	20	12
695	27	9	5.9	30.70	18	20	14	19	13
695(2)	30	17	6.1	24.92	29.7	16	11	16	16
696	39.5	6	5.8	27.09	29	22	18	21	12
697	23	10	5.1	28.10	19	11	6	21	15
698	36	14	6.3	23.39	27.9	19	15	4	16
700	29	8	6.0	28.48	20.6	nd[f]	nd	3	21
702	30.5	8	6.3	25.79	28.9	8	nd	5	16
703	33.3	6	6.5	24.94	30.2	tr[g]	nd	12	16
707	36.8	11	7.5	29.82	20.8	33	24	5	12
709	30.5	8	6.7	26.61	25	nd	nd	14	16
711	26.8	8	5.2	27.25	21.1	4	nd	16	17

a. Strongest uninterfered with line of substituted C_3S
b. Only measureable line of substituted C_2S
c. Strongest line
d. Lines of second and third intesity in C_3A
e. Uninterfered with line of fairly high intensity
f. nd = not detected
g. tr = trace

Table 9

Means, Standard Deviations, and Variances

Item	No. of Samples	Mean Value	Standard Deviation	Variance
Material insoluble in maleic acid, %	19	27.007	2.2973	5.27771
$C_4A_3\overline{S}$ (chart units at 3.76A)	19	24.426	3.732	13.924
Calcium Sulfate (anhydrite) (chart units at 3.495A)	19	30.068	14.943	223.30
C_3A (chart units at 1.904A)	19	15.910	9.983	99.653
Alite (chart units at 1.76A)	19	32.837	5.393	29.080
Belite (chart units at 2.88A)	19	10	2.944	8.666
Aluminoferrite (chart units at 7.2 - 7.4A)	19	15.790	5.369	28.825
Fineness, cm^2/g	19	4352	466	216,662
Restrained Expansion, %				
7-Days	19	0.052	0.0176	0.0003
28-Days	19	0.056	0.0203	0.0004
Compressive Strength, psi				
7-Days	19	4144	463	214,247
28-Days	19	5753	555	308,101
SO_3, %	19	6.02	0.688	0.47398

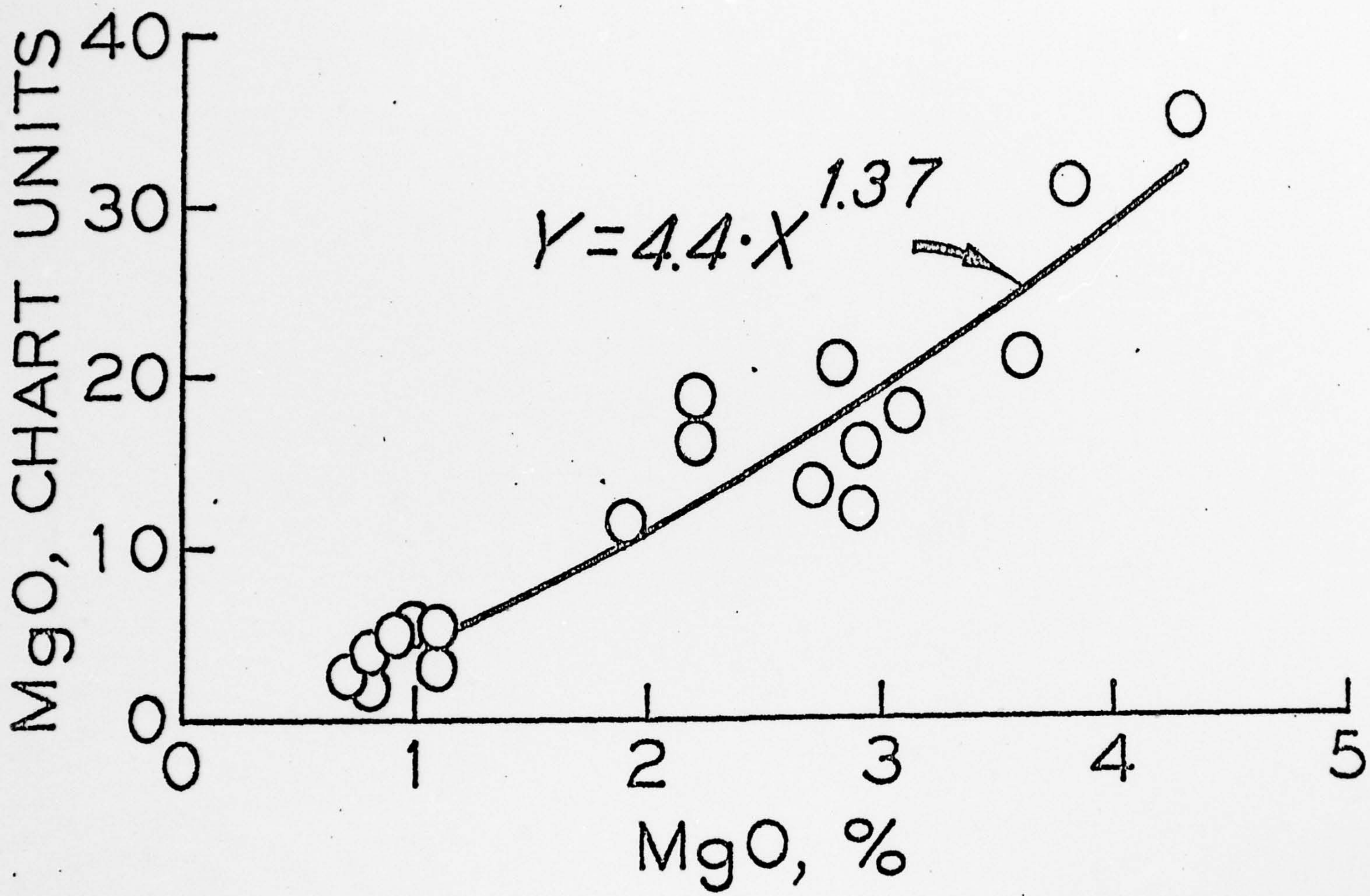

Figure 1. Least-Squares Curve Fit of MgO by Chemistry and by X-Ray Diffraction

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Hoff, George C

A look at Type K shrinkage-compensating cement production and specifications / by George C. Hoff, Katharine Mather. Vicksburg, Miss. : U. S. Waterways Experiment Station ; Springfield, Va. : available from National Technical Information Service, 1978.

ii, 23, [12] p. : ill. ; 27 cm. (Miscellaneous paper - U. S. Army Engineer Waterways Experiment Station ; C-78-2)

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References: p. 23.

1. Air content (Concrete). 2. Air entraining agents. 3. Compressive strength (Concrete). 4. Concrete drying shrinkage. 5. Expansive cement Type K. 6. Expansive cement concretes. 7. Expansive cements. 8. Mortars (Material). 9. Shrinkage compensating concretes. 10. Slump tests. 11. Specifications. 12. X ray diffraction. I. Mather, Katharine, joint author. II. United States. Army. Corps of Engineers. III. Series: United States. Waterways Experiment Station, Vicksburg, Miss. Miscellaneous paper ; C-78-2.

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